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ABRASIVE COATING MATERIAL  
[KENMAZAI TORYO]

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## Specifications

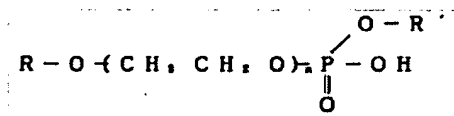
### 1. Title of the Invention

ABRASIVE COATING MATERIAL

### 2. Claim(s)

(1) An abrasive coating material characterized by being comprised of alumina, a polyurethane resin, an organophosphoric acid compound, and an organic solvent.

(2) The abrasive coating material of Claim (1) characterized by the organophosphoric acid compound being shown by the structural formula below.



(where R is an alkyl or aralkylallyl group; n is the number of added moles of ethylene oxide; R' is an H atom or  $\text{R-(CH}_2\text{CH}_2\text{O)}_n$  group).

(3) The abrasive coating material of Claim (2) characterized by the acid number and HLB value of the organophosphoric acid compound being 70 to 100 and 6.8 to 9.8, respectively.

### 3. Detailed Specifications

#### Field of Industrial Application

The present invention relates to an abrasive coating material useful in the manufacture of magnetic recording media.

#### Prior Art

In recent years, abrasive coating materials have been used widely in magnetic coating materials to manufacture magnetic recording media (video tapes, audio tapes, floppy disks, etc.). That is, less abrasives (alumina, chromium oxide, iron oxide red, etc.), which are indispensable

materials for improving the scanning durability characteristics of magnetic recording media, are used in the preparation of magnetic coating materials as powders; they are generally mixed with magnetic coating materials after making them into coating materials (into pastes or slurries) beforehand. The reason is because an increasingly higher dispersion of the abrasive is required and because there are various benefits since the DO (drop-out) of a finished video tape or the like is lower by adding an abrasive as a powder as the inner walls of a dispersing machine wear away the dispersing beads. Furthermore, a large amount of coating material is prepared at once for the abrasive coating material, stored for a long period of time, and then used, so it is needless to say that the dispersion stability, and of course, the dispersibility of the coating material, where the coating material attributes do not change over time, are strongly demanded. There were problems with conventional abrasive coating materials comprising only abrasive particles, a resin, and an organic solvent because the dispersion stability was poor and it was necessary to separate, clean, and redisperse the abrasive particles after long-term storage, but the conventional abrasive coating material could not be offered for practical use as their characteristics were not restored even after redispersing them.

#### Problems to be Solved by the Invention

An above-mentioned problem for abrasive coating materials included the fact that the dispersion stability of the abrasive particles, resin, and organic solvent composing the conventional abrasive coating material was not sufficient. Therefore, it was a problem to improve the dispersion

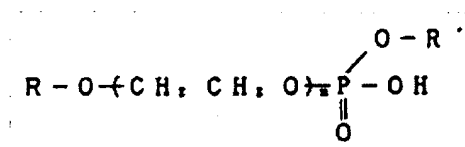
stability in order to enable long-term storage.

#### Means for Solving the Problems

In order to solve the above-mentioned problems, the abrasive coating material according to the present invention is composed of alumina, a polyurethane resin, an organophosphoric acid compound whose acid number is 70 to 110 and whose HLB value is 6.8 to 9.8, and an organic solvent.

#### Effects

The above organophosphoric acid compound is a phosphoric acid ester of a polyoxyethylene alkyl (or aralkylallyl) ether, which is a general ether-type nonionic activator. A typical chemical structure is as follows.



R: alkylallyl group

n: number of added moles of ethylene oxide

R': H atom or R(CH<sub>2</sub>CH<sub>2</sub>O)<sub>n</sub> group

The Al-O bonds of the alumina are hydrophilic, and consequently, compatibility with a compound having a highly polar functional group, such as an organophosphoric acid compound, is considered to be good for the organophosphoric acid compound comprising the above structural formula. Furthermore, in addition to having two long ethylene oxide chains, it is thought that, even if the ends are alkyl or alkylallyl groups, this organophosphoric acid compound easily bonds to the polyurethane resin. By using such a dispersant, the alumina can be dispersed with good efficiency while making the alumina into an abrasive coating material with a polyurethane resin, and moreover, an abrasive coating material having outstanding

dispersion stability where long-term storage is enabled without separation or sedimentation arising. The amount of the dispersant for the abrasive coating material is approximately several wt.% of the abrasive and the amount of the abrasive that is usually used is approximately several wt.% of the magnetic powder in a magnetic coating material; hence, the adverse effects of this dispersant on the physical characteristics of a magnetic recording medium is greatly reduced. When the formation of an abrasive coating material is performed from a polyurethane resin and organic solvent by using the dispersant for an alumina organophosphoric acid compound, the aforesaid phosphoric acid ester of a polyoxyethylene alkyl (or alkylallyl) ether is generally useful. In particular, the acid number and HLFB value of this dispersant are set to preferred ranges.

#### Practical Examples

A practical example of the present invention will now be described in detail.

#### Practical Example 1

The results of performing a sedimentation test to select a dispersant in a preliminary experiment are shown in Table 1. 0.3 g of a solution of a polyurethane resin (NV (weight ratio of nonvolatile constituent)=50.0%; solvent: methyl ethyl ketone (abbreviated "MEK," hereafter)) made by Takeda Chemical Industries, Ltd. (stock number: XE-21) were poured into a test tube (inner diameter: 13 mm; height: 150 mm). Next, 10.0 g of a mixed solvent (MEK/toluene/cyclohexane = 3/2/1) were poured into it. Furthermore, 1.0 g of highly pure  $\alpha$ -alumina (made by Sumitomo Chemical Co., Ltd.; stock number: AKP-50) and 0.05 g of a dispersant were added, the test tube was

stoppered so that the solvent did not evaporate therefrom, an ultrasonic dispersion was performed for thirty minutes, this was allowed to stand in a cold, dark place and a natural sedimentation test was performed. The results were more satisfactory when the organophosphoric acid compound (trade name: Plysurf) was added than in the comparative example in which a dispersant was not added, and there were no effects by other dispersants. That is, criteria for judging the dispersion stability three or more hours after sedimentation started was divided into three levels: extremely good (o), complete sedimentation plus markedly poor dispersibility (x), and midway between the above two levels ( $\Delta$ ). The quality of the dispersant was not clear.

Table 1

Sample No.	Dispersant Type, Stock No.	Judgment	Acid No.	HLB
1-1	No dispersant (comparative example)	x	-	-
1-2	Plysurf AL	$\Delta$	70-95	5.6
1-3	" A207H	o	71-85	7.1
1-4	" A208B	$\Delta$	160-185	6.6
1-5	" A208F	$\Delta$	160-180	8.7
1-6	" A210G	o	95-110	9.6
1-7	" A212C	$\Delta$	100-120	9.4
1-8	" A212E	$\Delta$	80-95	10.3
1-9	Myristic acid $\text{CH}_3(\text{CH}_2)_{12}\text{COOH}$	x	-	-
1-10	Toluic acid $\text{CH}_3\text{C}_6\text{H}_4\text{COOH}$	x	-	-
1-11	Lecithinic acid	x	-	-
1-12	Coconut oil	x	-	-

(Plysurf is a trade name of Daiichi Pharmaceutical Co., Ltd.)

Here, the acid number indicated the number of milligrams of KOH needed for neutralizing the free acid contained in 1 g of dispersant; it was measured in the following method.

3.00 g of Plysurf serving as the sample is accurately weighed into an Erlenmeyer flask and completely dissolved by adding about 40 mL of

neutral solvent. Several drops of a 1% ethyl alcohol solution of phenolphthalein are added as the indicator, a measurement with 0.5N potassium hydroxide is, and the endpoint is obtained where the color of the indicator (light pink) persists for one minute. The acid number is calculated according to the following formula,

Acid number =  $(56.11 \times 0.5 \times F \times \text{no. of mL measured}) / (3.00 (\text{no. of grams of sample}))$

provided that **F** is the titer of the potassium hydroxide solution.

Although the HLB value was a value indicating the hydrophilic/lipophilic balance of the dispersant, it was calculated next according to an equation according to Kawakami for nonionic surfactants.

$$\text{HLB} = 7.0 + 11.7 \log (M_w/M_o)$$

$M_w$ : molecular weight of hydrophilic group;  $M_o$ : molecular weight of lipophilic group

The organophosphoric acid compound is effective on the dispersion stability, as shown in Table 1.

#### Practical Example 2

The abrasive coating material was prepared by using a 1.6 L alumina pot (the dispersing beads were an alumina number with a 5 mm diameter). Dispersion was performed for thirty minutes at a rotating speed of 60 rpm. Measurement of the average particle diameter (median) of the alumina in the abrasive coating material was performed in a method for evaluating the dispersibility of this alumina by using the same AKP-50 as in Practical Example 1 as the abrasive. Then it was measured at the following conditions using a centrifugal sedimentation-type automatic particle size distribution



measuring device made by Horiba, Ltd. (CAPA-300).

The range of the particle diameter to be measured (alumina density: 3.99 g/cm<sup>3</sup>; diluted MEK solvent specific gravity: 0.81 g/cc; viscosity of diluted MEK solvent: 0.44 cps) was set at 2.0 to 0.1 μm (0.1 μm spacing) and the centrifugal sedimentation rotating speed was set to 1,500 rpm. A sedimentation experiment was performed to evaluate the dispersion stability according to the sedimentation experiment of the abrasive coating material prepared next.

That is, 1.15 g of the abrasive coating material serving as the sample was weighed as shown below, mixed with 10.3 g of a mixed organic solvent (MEK/toluene/cyclohexanone; =3/2/1 by weight ratio), and collected in a test tube (inner diameter: 13 mm; height: 150 mm). The test tube was stoppered so that the solvent did not evaporate, an ultrasonic dispersion was performed for five minutes, this was allowed to stand in a cold, dark place, and a natural sedimentation test was performed. The results of are displayed in five levels of a visual judgment of the dispersion stability: from very good dispersion stability (DC [double circle]) to complete stability is poor and there is immediate particle sedimentation (××). That is, the dispersion stability was judged (DC, o, Δ, ×, and ××) upon comparison with the 5-level minimum acceptance sample fabricated in advance.

The abrasive coating material sample in this Practical Example 2 is charged with the following constituents:

Alumina (made by Sumitomo Chemical Co., Ltd.: stock no.: AXP-50):  
371 g

Dispersant (made by Daiichi Pharmaceutical Co., Ltd.; Plysurf (trade name)): 18.55 g

Solution (NV = 50.0%; MEK solution) of polyurethane resin (made by Takeda Chemical Industries, Ltd.; stock no.: XE-21): 111.3 g

Mixed organic solvent (MEK/toluene/cyclohexanone (= 3/2/1 by weight ratio)): 56.3 g

These materials were charged in one batch. The obtained results are shown in Table 2.

Table 2

Sample No.	Dispersant Type, Stock No.	Median value ( $\mu\text{m}$ )	Sedimentation Time (hr.)			
			1	10	20	30
2-1	No dispersant (comparative example)	0.33	x	x	x	x
2-2	Plysurf AL	0.29	o	$\Delta$	x	x
2-3	" A207H	0.25	DC	DC	DC	DC
2-4	" A208B	0.29	o	$\Delta$	x	x
2-5	" A208F	0.29	$\Delta$	$\Delta$	x	x
2-6	" A210G	0.25	DC	DC	DC	DC
2-7	" A212C	0.29	$\Delta$	$\Delta$	x	x
2-8	" A212E	0.29	$\Delta$	$\Delta$	x	x

It is evident that the median, 0.25  $\mu\text{m}$ , is approximately the same as the size of a primary particle of the abrasive, AKP-50, and it disperses sufficiently. Of the stock number of Plysurf, A207H and A210G are excellent in particular upon observing an electronic microscopic photograph thereof. It is thought that the reason is because a balance between the acid number and HLB value of the alumina and polyurethane is kept. That is, it is understood that the acid number is good between 70 and 110, and concurrently, the HLB is good between 6.8 and 9.8.

### Practical Example 3

In order to prepare a large amount of abrasive coating material, a 50 L pebble mill (model: PBM-50; dispersing beads: 60 kg of 25 mm diameter alumina beads) made by Inoue Manufacturing Co., Ltd., which was a ball mill-type dispersion machine coated on the inside with alumina, was used to perform a dispersion test. It is charged with the materials, as shown below, as a batch and the dispersion time was set to sixty hours.

Alumina (made by Sumitomo Chemical Co., Ltd.; stock no. AKP-50):  
15,000 g

Dispersant (Plysurf, made by Daiichi Pharmaceutical Co., Ltd.): 225 g

Solution (NV = 50.0%; MEK solution) of polyurethane resin (made by Takeda Chemical Industries, Ltd.; stock no.: XE-21): 4,500 g

Mixed organic solvent (MEK/toluene/cyclohexanone (=3/2/1 by weight ratio)): 2,275 g

In this practical example, the amount of the dispersant with respect to alumina that was changed to 5 wt.% in Practical Examples 1 and 2 was set to 1.5 wt.%.

The measurement of the median and the sedimentation test method were performed in the same method as in Practical Example 2. The results are shown in Table 3.

Table 3

Sample No.	Dispersant Type, Stock No.	Median value (μm)	Sedimentation Time (hr.)			
			1	10	20	30
3-1	No dispersant (comparative example)	0.29	Δ	Δ	×	×
3-2	Plysurf A207H	0.26	DC	DC	DC	DC
3-3	" A210G	0.25	DC	DC	DC	DC

As evident from the results in Table 3, the organophosphoric acid compound is useful as the dispersant, and in particular, the phosphoric acid ester of the polyoxyethylene alkyl (or aralkylallyl) ether having the aforesaid structural formula, as represented by Plysurf (trade name) made by Daiichi Pharmaceutical Co., Ltd. is useful. The effects of the dispersant on dispersion stability therein are remarkable according to the acid number and the HLB thereof. That is, an acid number of 70 to 100 and an HLB of 6.8 to 9.8 are desirable. It is clear that A 207H and A 210G, which satisfy the value in each range, exhibit remarkable effects.

Six 6 months after preparing the coating materials, no problem in the characteristics of a prototype videotape manufactured was confirmed by using the abrasive coating materials actually prepared in Test Nos. 3-2 and 3-3. That is, no insufficiency in dispersion stability of the abrasive coating material arises. Changes in the amount of alumina in the composition, aggregation, an abnormal DO [misspelled in source as 'DC'] (drop-out), poor steel characteristics, and the like did not develop.

There were no substantial changes in the characteristics of the coating material of the abrasive coating materials set and stored in a cold, dark place stoppered firmly and set aside for six months after preparation.

#### Advantages of the Invention

While performing dispersion and preparation of the abrasive coating material from alumina and a polyurethane resin as mentioned above, the organophosphoric acid compound is useful as the dispersant, and in particular, the phosphoric acid ester having a polyoxyethylene alkyl (or aralkylallyl) ether structure is superior. Alumina is the mainly used abrasive in magnetic

recording media, and in particular, video tapes, and highly pure  $\alpha$ -alumina is used widely in high-performance video tapes. On the other hand, a polyurethane resin is indispensable as a binder resin, and it is needless to say that a superior abrasive coating material with dispersibility and satisfactory dispersion stability may be prepared by using a suitable dispersant, as in the present invention, which may be applied widely in other fields besides the manufacture of magnetic recording media. A superior abrasive coating material may be prepared by selecting in addition to alumina, chromium oxide and iron oxide red, and various suitable dispersants and resins as well as organic solvents.